

# PATENT ABSTRACTS OF JAPAN

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## (54) CURABLE COMPOSITION

(57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a curable composition giving a cured product which ensures practical curability and recovery properties and exhibits mechanical properties of high strength and high elongation.

**SOLUTION:** The curable composition is characterized by comprising (A) an organic polymer having at least one silicon-containing group which has a hydroxy or hydrolyzable group bonded to the silicon atom and is crosslinkable by forming a siloxane bond, and (B) one or more metal salts of a carboxylic acid selected from calcium carboxylate, vanadium carboxylate, iron carboxylate, titanium carboxylate, potassium carboxylate, barium carboxylate, manganese carboxylate, nickel carboxylate, cobalt carboxylate, and zirconium carboxylate.



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2. The word "drawings" allows the word which can not be translated.

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[Field of the Invention]

[0001]

In this invention, it has the hydroxyl group or hydroxylic basis combined with the silicon atom, and a siloxane bond is formed. Therefore, it is related with the hardenability constituent containing the organic polymer which has a silicon content group (hereinafter a "reactive silicon group") which can construct a bridge.

[Background of the Invention]

[0002]

It is known that the organic polymer which contains at least one reactive silicon group in a molecule has the following character in which construct a bridge by formation of the siloxane bond accompanied by the hydrolysis reaction of a reactive silicon group, etc., and a rubber-like hardened material is obtained with hygroscopic surface moisture etc. also in a room temperature.

[0003]

In the polymer which it has, these reactive silicon groups a polyoxysilane series polymer and a polysiloxane series polymer. It is indicated by JP-S52-73988A, JP-H5-12572A, JP-H3-72571A, JP-S63-6003A, JP-S63-6041A, JP-H1-38407A, JP-H6-231758A, etc.

Especially a polyoxysilane series polymer and a polysiloxane system polymer are already produced industrially, and are widely used for uses, such as a sealing material, adhesives, and a paint.

[0004]

When an organic polymer is a saturated hydrocarbon system polymer especially, since it excels in heat resistance, a water resisting property, weatherability, etc. if the hardened material obtained is used for a structural sealing material, the sealing material for multiple glass, etc. it is effective. The isobutylene system polymer which has a repeating unit which furthermore originates in isobutylene 80% of the weight or more in a total amount is preferred as a sealant aiming its moisture proof from especially the thing to excel in low moisture permeability and low gas permeability.

[0005]

Such a sealing material generally fills up the joined part and cures between various members, the effectiveness to use part ranging from being used to a wide field of time factor to be called for.

[0006]

Amine compounds, such as lauryl amine, are used together as a co-catalyst in many cases for the purpose of a silanol condensation catalyst being used for bridge construction hardening of an organic polymer which, on the other hand, has such a reactive silicon group, and also accelerating a condensation reaction. As such a silanol condensation catalyst, divalent tin, such as octylic acid (2-ethylhexanoic acid), the tin series catalyst represented by trivalent tin, such as dibutyltin bisacrylate/lactonate, is used widely, and when the movement of the part which pieces especially a sealing material is large, since the hardened material which has stability is obtained, many divalent tin is used.

[0007]

However, when octylic acid tin which is divalent tin, for example is used together with the amine compound of a co-catalyst and is used as a curing catalyst of the (A) ingredient, the intensity and elongation of a hardened material which are acquired may be insufficient, and an improvement of the further physical properties is desired.

[0008]

Although most is a dibutyltin type, the latter tetravalent tin series catalyst being used now. While the hardened material obtained has emollient, i.e., having the character to follow to the long-term stress change from the outside and fast curability, it is broadly used from the ability to apply as a curing catalyst of a liquid mold-curing nature constituent. However, since toxicity in case a small amount of the material contains in this is regarded as questionable, development of the silanol condensation reaction of a non-tin series is desired.

[0009]

In JP-S35-2765B, JP-S32-3424B, JP-S35-9639B, JP-S37-3271B, etc. As a curing catalyst of an organopolysiloxane constituent, the carboxylic acid metal salt of various kinds of non-tin is indicated with carboxylic acid tin. As an example in which the carboxylic acid metal salt of non-tin is used for the curing catalyst of the constituent which, on the other hand, contains the organic polymer which has a reactive silicon group, although there were carboxylic acid lithium (JP-H5-39428A, JP-H6-12860A) and carboxylic acid cerium (JP-2000-313814A), there was no example broadly examined in various carboxylic acid metal salt until now.

[Patent documents 1] JP-S52-73988A

[Patent documents 2] JP-H5-12572A

[Patent documents 3] JP-H3-72571A

[Patent documents 4] JP-S63-6003A

[Patent documents 5] JP-S63-6041A

[Patent documents 6] JP-H1-38407A

[Patent documents 7] JP-H6-231758A

[Patent documents 8] JP-S35-2765B

[Patent documents 9] JP-S35-9639B

[Patent documents 10] JP-S37-3271B, etc.

[Patent documents 11] JP-S32-3424B

[Patent documents 12] JP-H5-39428A

[Patent documents 13] JP-H6-12860A

[Patent documents 14] JP-2000-313814A

[Description of the Invention]

[Problem(s) to be Solved by the Invention]

[0010]

The purpose of this invention is as follows.

Have practical hardenability and stability with a good hardened material obtained.

Provide a hardenability constituent useful as the structural sealing material asked especially for high durability, the sealing material for multiple glass, the moisture seal material used for an electric electronic component, adhesives, etc. from revealing the outstanding intensity and elongation.

[Means for Solving the Problem]

[0011]

In order that this invention persons may solve such a problem, as a result of inquiring, as a curing catalyst of this (A) ingredient Carboxylic acid calcium, Carboxylic acid vanadium, carboxylic acid iron, carboxylic acid titanium, Carboxylic acid potassium, By using one or more sorts of carboxylic acid metal salt chosen from carboxylic acid sodium, carboxylic acid potassium, carboxylic acid calcium, carboxylic acid vanadium, carboxylic acid iron, carboxylic acid titanium, carboxylic acid potassium, and carboxylic acid calcium, Sealing moderate hardenability and stability, it finds out revealing high intensity and the physical properties of high elongation compared with a case where octylic acid tin is used as a curing catalyst, and came to complete this invention.

[0012]

Namely, this invention has a hydroxyl group or a hydroxylic basis combined with the (A) silicon atom. It is related with a hardenability constituent containing one or more sorts of carboxylic acid metal salt chosen from an organic polymer, (B) carboxylic acid iron, and carboxylic acid cobalt which have at

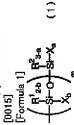
least one silicon content group which can construct a bridge by forming a siloxane bond.

[0013]

An amine compound is related with the aforementioned hardenability constituent which becomes as an essential ingredient as a (C) ingredient.

[0014]

As a desirable embodiment, a number average molecular weight is within the limits of 500-50,000, and as an organic polymer of the (A) ingredient is a general formula at an end and/or a side chain of a main chain (1):



[Formula 1]

[0016]

(R<sup>1</sup> and R<sup>2</sup> among a formula) They are an alkyl group of the carbon numbers 1-20, an aryl group of the carbon numbers 6-20, an aralkyl group of the carbon numbers 7-20, or (R<sup>3</sup>)<sub>3</sub>SiO independently, respectively. —(R<sup>3</sup>)— respectively — the substitution of the carbon numbers 1-20, or an unsubstituted hydrocarbon group — it is — it is the tin ORGANO group below. X is a hydroxyl group or a hydroxyloxy base independently, respectively, a 0, 1, 2, or 3, 4, 5, 6, 7, 8, 9, and a 9 are not simultaneously set to 0 in the integer of 0, or 19. It is related with the hardenability constituent given in said either having one or more hydroxyl alkyl groups per molecule independently.

[0017]

It is related with the aforementioned hardenability constituent characterized by X being an alkoxy group as a desirable embodiment.

[0018]

As a desirable embodiment, the organic polymer of the (A) ingredient is related with a hardenability constituent given in said either which is a polyoxyalkylene series polymer and/or a saturated hydrocarbon system polymer.

[0019]

As a desirable embodiment, said saturated hydrocarbon system polymer is related with the aforementioned hardenability constituent which is a polymer having a repeating unit resulting from isobutylene 50% of the weight or more in a total amount.

[0020]

As a desirable embodiment, carboxylic acid iron of the (B) ingredient and carboxylic acid cobalt are related with a hardenability constituent given in said either which uses as the main ingredients carboxylic acid metal salt expressed with the general formula (4), (5), and (11), respectively.

[0021]

(The inside R of a formula is substitution or an unsubstituted hydrocarbon group which has the third class carbon or quaternary carbon which adjoins a carbonyl group, and may include a carbon carbon double bond.)

[0022]

As a desirable embodiment, carboxylic acid metal salt of the (B) ingredient is related with a hardenability constituent given in said either which is the carboxylic acid metal salt in which the melting point has an acid radical of carboxylic acid which is 65 °C or less.

[0023]

As a desirable embodiment, a carbon number in which carboxylic acid metal salt of the (B) ingredient contains carbon of a carbonyl group is related with a hardenability constituent given in said either which is the carboxylic acid metal salt which has an acid radical of carboxylic acid which is 2-17.

[0024]

As a desirable embodiment, carboxylic acid metal salt of the (B) ingredient is related with a hardenability constituent given in said either which is metal salt of a carboxylic acid group content compound chosen from 2-ethylhexanoic acid, neo decanoic acid, or naphthoic acid.

[0023]

It is related with a hardenability constituent given in said either containing the (B) ingredient of quantity which serves as 0.005 ~ 10 weight section by metallic element conversion contained in the (B) ingredient to (A) ingredient 100 weight section as a desirable embodiment.

[0024]

It is related with a hardenability constituent given in said either containing the (B) ingredient of quantity which serves as 0.005 ~ 10 weight section by metallic element conversion contained in the (B) ingredient to (C) ingredient 0.01 ~ 20 weight section to (A) ingredient 100 weight section as a desirable embodiment.

[Best Mode of Carrying Out the Invention]

[0025]

Hereafter, this invention is explained in detail.

[0026]

Restriction in particular does not have a principal chain skeleton of an organic polymer which has a reactive silicon group used for this invention, and it can use a thing with various kinds of principal chain skeletons.

[0027]

Specifically A polyoxyethylene, polyoxypropylene, polyoxybutylene, polyoxy tetramethylene, a polyoxyethylene polyoxypropylene copolymer, or polyoxyalkylene series polymers, such as a polyoxypropylene polyoxy butylene copolymer, An ethylene-propylene system copolymer, The copolymer of polyisobutylene, isobutylene, isoprene, etc., polycyclopentene, A copolymer with polystyrene, isoprene or butadiene, acrylonitrile, styrene, etc., hydrocarbon system polymers, such as a hydrogenation polyolefin system polymer produced by hydrogenating polyolefins or these polyolefin system polymer; Condensation with dibasic acid, such as adipic acid and BPOC. Or the polyester system polymer obtained by the ring opening polymerization of lactone. The polyarylether system polymer obtained by carrying out the radical polymerization of aryl ether monomers, and butoxyethyl acrylate and butyl acrylate and butyl acrylate, vinyl acetate, acrylonitrile, methyl methacrylate, styrene, etc.; A vinyl monomer is polymerized to said organic polymer. Graft polymer, obtained.

Polyamide system polymer: Nylon 610 by the condensation polymerization of the nylon 6 by the ring opening polymerization of epsilon caprolactam, hexamethylenediamine, and sebacic acid. Nylon 66 by the condensation polymerization of adipic acid and hexamethylenediamine, and sebacic acid. Nylon 11 by the condensation polymerization of apalmon-undecanedioic acid, and condensation polymerization is carried out from polyamide system polymer, for example, bisphenol A, and the carbonyl chlorides which have a two or more-ingredient ingredient among Nylon 12 by the ring opening polymerization of epsilon-aminic RAUO lactam, and the above-mentioned nylon, such as copolyamide. The polycarbonate system polymer manufactured, a diallyl phthalate system polymer etc. are illustrated.

[0028]

Since acquisition and manufacture are easy among polymers with the above-mentioned principal chain skeleton, a polyoxyalkylene series polymer, a hydrocarbon system polymer, a polyester system polymer, a vinyl system copolymer, a polycarbonate system polymer, etc. are preferred. A saturated hydrocarbon system polymer, and a polyoxyalkylene series polymer and a vinyl system copolymer have a comparatively low glass transition temperature, and their hardened material obtained is especially preferred from excelling in cold resistance.

[0029]

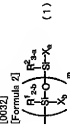
As a saturated hydrocarbon system polymer which has a reactive silicon group used for this invention, the reactive silicon group content saturated hydrocarbon system polymer derived from saturated hydrocarbon system polymers, such as polyisobutylene, hydrogenation polybutadiene, and hydrogenation polystyrene, is raised.

[0030]

The reactive silicon group contained in the organic polymer which has a reactive silicon group is a basis which can construct a bridge by formation of the siloxane bond which is a reaction which has

the hydroxyl group or hydrolytic basis combined with the silicon atom, and is accelerated with the carboxylic acid metal salt which is the (B) ingredient.

As a reactive silicon group, it is a general formula (1):



[0003] (R<sup>1</sup> and R<sup>2</sup> among a formula) They are an alkyl group of the carbon numbers 1-20, an aryl group of the carbon numbers 6-20, an allyl group of the carbon numbers 7-20, or (R<sup>3</sup>)  $\gamma$ -SO independently, respectively. (R<sup>3</sup>) respectively — independent — substitution of the carbon numbers 1-20, or an unsubstituted hydrocarbon group — it is — it is the Tori ORGANO alloy group shown. X is a hydroxyl group or a hydrolytic basis independently, respectively, a is 0, 1, 2, or 3, b is 0, 1, or 2, and a and b are not simultaneously set to 0, m — an integer of 0, or 1-19 — it is — a basis expressed is raised.

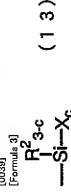
[0004] It is not limited but what is necessary is just a conventionally publicly known hydrolytic basis especially as a hydrolytic basis. A basis for which a hydrogen atom, a halogen atom, an alkoxo group, an aryloxy group, a KETOKISHI mate group, an amino group, an amide group, an acid-amino group, an aminoxy group, a sulfinyl group, an alkenyloxy group, etc. are generally used concrete, for example is raised.

[0005] Among these, although an alkoxo group, an amide group, and an aminoxy group are preferred, a point of hydrolysis nature being quiet and being easy to deal with it to especially an alkoxo group is preferred.

[0006] A hydrolytic basis and a hydroxyl group can be combined with one silicon atom in the 1-3 ranges, and (a-1) group has 1-5 preferred ranges. When a hydrolytic basis and a hydroxyl group join together in [two or more] a reactive silicon group, they may be the same and may differ.

[0007] Although a silicon atom which forms a reactive silicon group is one or more pieces, in the case of a silicon atom connected by a siloxane bond etc., it is preferred that they are 20 or less pieces.

[0008] In particular, it is a general formula (13):



[0009] (R<sup>2</sup> and X are the same as the above among a formula) c is an integer of 1-3. Since the reactive silicon group expressed is easy to resolve, it is preferred.

[0010] In the above-mentioned general formula (13), Tori alkoxo silyl groups, such as a methoxy group, an ethoxy group, a propoxy group, and an isopropoxy group, is mentioned for X as an example in case c is 3. In the above-mentioned general formula (13), as an example of R<sup>2</sup> in case c is not 3, for example,

cycloalkyl groups, such as silyl groups, such as a methyl group and an ethyl group, and a cyclohexyl group, Aralkyl groups, such as aryl groups, such as a phenyl group, and benzyl, the Tori ORGANO alloy group R<sup>2</sup> is indicated to be by  $\gamma$ -SO— which is a methyl group, a phenyl group, etc. (R<sup>2</sup>), etc. are mentioned. In these, the point that the activity of a hydrolysis reaction is high to especially a methyl group is preferred.

[0042] As more concrete illustration of a reactive silicon group, a trimethoxysilyl group, a triethoxysilyl group, a triisopropoxysilyl group, a dimethoxymethyl silyl group, a diethoxymethyl silyl group, and a diisopropoxymethyl silyl group are mentioned. Its activity is high, and since especially the trimethoxysilyl group can reduce quantity of carboxylic acid metal salt which is the (B) ingredient, it is preferred.

[0043] What is necessary is just to perform introduction of a reactive silicon group by a publicly known method. That is, the following methods are mentioned, for example.

(b) Make an organic compound which has an active group and an unsaturation group which allow reactivity to an organic polymer which has functional groups such as a hydroxyl group, in a molecule to a functional group and obtain a reactive silicon group. Or an unsaturation group and a functional group are made to react independently by a condensation reaction with an unsaturation group content epoxy compound. Subsequently, hydroxyls which have a reactive silicon group is made to act on an acquired reactant, and it hydrolyzes.

[0045] (\*\*) Make a compound which has a sulfinyl group and a reactive silicon group react to an organic polymer containing an unsaturation group produced by making it be the same as that of the (b) method.

[0046] (\*\*) Make a compound which has a functional group and a reactive silicon group which allow reactivity to an organic polymer which has functional groups, such as a hydroxyl group, an epoxy group, and an isocyanate group, in a molecule to this functional group react.

[0047] A method of making a compound which has a polymer, an isocyanate group, and a reactive silicon group which has a hydroxyl group react to an end a method of (b) or among (\*\*) is from points, like a manufacturing cost becomes low in the above method is preferred.

(b) As an example of a hydroxyl compound used in a method, for example, triethoxysilane, methyl diethoxysilane, dimethyldichlorosilane, Halogenated Silane like vinylchlorosilane, Trichlorosilane, trichloroethoxysilane, Methyl octadecylsilane, The alkyl groups like phenyl, hexadecyl, octadecyl, etc. are mentioned. The alkyl groups like a phenyldichlorosilane, although the KETOKISHI mate silanes like bisdimethyl KETOKISHI methoxymethyls and bis(cyclohexyl KETOKISHI methoxymethyls) are raised. It is not limited to these. Among these, halogenated Silane and alkoxy silanes are especially preferred from points, like availability and hydrolysis reaction nature are high.

[0048] (\*\*) Although a method of introducing into an unsaturation binding site of an organic polymer a compound which has a sulfinyl group and a reactive silicon group as a synthetic method by a radical addition reaction under a radical initiator and/or radical source-of-release existence, for example, etc. are mentioned, it is not limited to particular. As an example of a compound of having said sulfinyl group and a reactive silicon group, for example, although gamma-mercaptopropyltrimethoxysilane, gamma-mercaptopropylmethyl dimethoxysilane, gamma-mercaptopropyl triethoxysilane, gamma-mercaptopropylmethyl diethoxysilane, etc. are raised, it is not limited to these.

[0050] Although a method etc. which are shown in JP-40-47805A are mentioned, for example as a method of making a compound which has a polymer, an isocyanate group, and a reactive silicon group which have a hydroxyl group reacting to an end among synthetic methods, it is not limited in





polymer as oxy metal groups, such as  $-ONa$  and  $-OK$ , first, for example:



Halogen atoms, such as a chlorine atom, a bromine atom, and iodine atoms, and  $R^1$  are among [Type, and Y is  $-R^2$ ,  $-R^3-OO-$ , or  $-R^3-SO-$  ( $R^2$  is a divalent hydrocarbon group of the carbon numbers 1-20, and) an alkylene group, a cyclo alkylene group, an alkylene group, and an aralkylene group — it is desirable — by a divalent organic group shown. — It can obtain by making an organic halogenated compound shown by especially a carbon numbers 1-10 being preferred) react.

[0094]

As a method of using terminal hydroxyl groups of an end hydroxy hydrogenation polybutadiene system polymer as a oxy metal group, A method of making it react to alkaline-water oxides, such as Na, metal alkoxide-NaOH like metal hydride-NaOCH<sub>3</sub> like alkaline metal-NH like K, and KOH, etc. is raised.

[0095]

Although an end olefin hydrogenation polybutadiene system polymer with the almost same molecular weight as an end hydroxy hydrogenation polybutadiene system polymer used as a starting material is obtained in said method. Before making an organic halogenated compound at a given time, (17) react to obtain a polymer of the amount of Polymer Division more than 1 at the time such as a methylene chloride, chloroform, and carbon tetrachloride. If it is made to react to an organic halogenated compound which can increase a molecular weight if it is made to react to a multivalent organic halogenated compound which contains two or more halogen in one molecule, and is shown by a general formula (17) after that. A hydrogenation polybutadiene system polymer which is the amount of Polymer Division more, and has an olefin group at the end can be obtained.

[0096]

As an example of an organic halogenated compound shown by said general formula (17), For example, an allyl chloride, an allyl star's picture, vinyl(chloromethyl) benzene, allyl(chloromethyl) benzene, allyl (bromomethyl) benzene, Although allyl (chloromethyl) ether, allyl(chloromethoxy) benzene, 1-butenyl (chloromethyl) ether, 1-hexenyl(chloromethoxy) benzene, allyloxy (chloromethyl) benzene, etc. are raised. It is not limited to them. Among these, an allyl chloride is cheap, and since it moreover reacts easily, it is desirable.

[0097]

Introduction of a reactive silicon group to said end olefin hydrogenation polybutadiene system polymer may be manufactured by carrying out an addition reaction using a platinum system catalyst in a hydrocarbon compound like a case of an isobutylene system polymer which has a reactive silicon group in molecular chain terminals.

[0098]

When a saturated hydrocarbon system polymer which has a reactive silicon group as mentioned above does not contain substituent which is a saturated bond which is not an aromatic ring in a molecule. Compared with a conventional rubber system polymer like an

organic polymer, a polyoxalkylene system polymer which has an unsaturated bond, its weatherability becomes good remarkably. Since this polymer is a hydrocarbon system polymer, its water resisting property is good, and a low hardened material of humidity permeability is obtained.

[0099]

An organic polymer which has these reactive silicon groups may be used alone, and may be used together two or more sorts. Specifically, a polyoxalkylene series polymer which has a reactive silicon group, a saturated hydrocarbon system polymer which has a reactive silicon group, a vinyl-base polymer which has a reactive silicon group, and an organic polymer which blends two or more sorts chosen from a group, \*\* and others, can also be used.

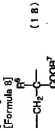
[0100]

A manufacturing method of an organic polymer which blends a polyoxalkylene series polymer which has a reactive silicon group, and a vinyl-base polymer which has a reactive silicon group. Although indicated by JP 69-122651A, JP 63-112662A, JP 68-172651A, JP 63-116763A, etc., it is not limited to in particular these.

[0091]

A desirable example has a reactive silicon group and a chain is a following general formula substantially (18):

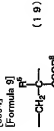
[0092]



[0093]

the acrylic ester monomer unit which has an alkyl group of the carbon numbers 1-8 expressed with (a) hydrogen atom or a methyl group, and  $R^2$  show the alkyl group of the carbon numbers 1-8 among a formula, as for  $R^2$  (meta-), and following general formula (19):

[0094]



[0095]

(— the heads of a formula and  $R^3$  — the same —  $R^3$  shows a with a carbon numbers of ten or more alkyl group —) to the copolymer which consists of an acrylic ester monomer unit which has a with a carbon numbers of ten or more expressed alkyl group (meta-). It is the method of blending and manufacturing the polyoxalkylene series polymer which has a reactive silicon group. Acrylic acid (meta) expresses acrylic acid and/or methacrylic acid with the above-mentioned excessive form.

[0096]

As  $R^1$  of said general formula (18) — the carbon numbers 1-8 of a methyl group, an ethyl group, a propyl group, n-butyl group, t-butyl group, a 2-ethylhexyl group, etc. — desirable — 1-4 — an alkyl group of 1-2 is raised still more preferably. An alkyl group of  $R^1$  may be independent and may be mixed two or more sorts.

[0097]

As  $R^2$  of said general formula (18) — two or more carbon numbers of a lauryl group, a tridecyl group, a eeryl group, a stearyl group, a behenyl group, etc. — usually — 10-30 — a long-chain alkyl group of 10-20 is raised preferably. Like a case of  $R^1$ , an alkyl group of  $R^2$  may be independent and may be mixed two or more sorts.

[0098]

Although a chain of this vinyl system copolymer consists of a monomeric unit of a formula (18) and a formula (19) substantially, a "real target", here means that the sum total of a monomeric unit of a formula (18) which exists in this copolymer, and a formula (19) surpasses 50 % of the weight. The sum total of a monomeric unit of a formula (18) and a formula (19) is 70 % of the weight or more preferably.

[0099]

From a point of compatibility with a polyoxalkylene series polymer, as for an abundance ratio of a monomeric unit of a formula (18), and a monomeric unit of a formula (19), 95:5-40:60 are preferred at a weight ratio, and 90:10-40:60 are still more preferred.

[0100]

As monomeric units other than a formula (18) which may be contained in this copolymer, and a formula (19), For example, carboxylic acid groups, such as acrylic acid and methacrylic acid, acrylamide, Amide groups, such as methacrylamide, N-methylacrylamide, and N-methylmethacrylamide, Epoxy groups, such as glycidyl acrylate and glycidyl methacrylate, Dimethylamino ethyl acrylate, diethylamino ethyl methacrylate, A monomer containing amino groups.









hardenable constituent of this invention, for example, was described above is blended, it kneads under ordinary temperature or heating using a mixer, a roll, a kneader, etc., or an ingredient is dissolved using a little suitable solvents, and a usual method of mixing may be adopted. A liquid type, two-component type, or many liquid type compound can also be built and used by combining these ingredients suitably.

[0136]

[0,136]  
If a hardenability constituent of this invention is exposed into the atmosphere, by operation of moisture, it will form network structure in three dimensions, and will harden it to a solid which has rubber-like elasticity.

[0137]

In a hardenable constituent of this invention, various additive agents can be added if needed. Other curing catalysts as examples of said additive for example (for example, tetraivalent tin, dibutyltin compounds) are known in the art.

Adhesive grant agents which adjust the ttractive characteristics of a hardened material to generate, such as a physical-property regulator and a silane coupling agent. An antioxidant, radical inhibitor, an ultraviolet-ray absorber, a metal deactivator, anti-oxidant light stabilizer, the Lym generator, an ultraviolet ray absorbent, a radical scavenger, etc., may also be included.

The system peroxide decomposition agent, lubricant, paints, a foaming agent, a photo-setting resin, a thermosetting resin, a photopolymerization initiator, a crosslinking agent, etc. are also

[0138]

[0136]  
An example of such an additive is indicated in each gazette of JP.4-69659.B, JP.7-108928.B, JP.63-254149.A and JP.64-22904.A, for example.

[0139]

Since a hardenable constituent of this invention has outstanding weatherability, heat resistance, flame retarding property, and electric insulation resulting from a principal chain skeleton and it has high intensity and a physical-mechanical improvement effect of high elongation, it can use conveniently for various insulation materials, such as electric elongation component materials, such as a structural electric insulating material, a sealing material for multiple glass, a solar cell near-face sealing agent, protection of insulating electric wire, material for cables, a binder, adhesives, a paint, infiltration, a coating material, a sealing agent for paint prevention / water proof, etc. It is useful especially when used for the production of a hardenable plastic, a hardenable plastic compound, or a sealing material for bonding.

### Example

[0140] Although working example and a comparative example explain this invention concretely below, this invention is not limited to this.

[0141]

(194) For example 1-4, comparative example 1) and 2) as opposed to isobutylene system polymer (Kasei Corp. melk. trade name EP005S;isobutylene system polymer / paraffin series process oil = 100/50) 150 weight section which has a reactive silicon polymer as an ingredient. Epoxy resin (product [ made from Oil recovery Shell Epoxy ] trade name Epiocast 828) 5 weight section. Hydrogenation alpha-olefin oligomer (product [ made from Idemitsu Petrochem ] , trade name PA06004) alpha-olefin oligomer. Colloid calcium carbonate (Maruo Calcium Co., Ltd. melk. trade name SHRETTUSU 200) 50 weight section. Colloid calcium carbonate (Maruo Calcium Co., Ltd. melk. trade name MC-50) 50 weight section. Heavy-calcium carbonate (product [ made from Shirotsu Calcium ] , trade name SOUTON 3200) 40 weight section. Photo-setting resin (Taogosei melk. trade name ARONIKUSU M-305) 3 weight section. 392) 1) Benzoinzitol system ultraviolet ray absorbent (Chiba-Gyō Japan melk. trade name SANPELUS-7770) weight section. Hindered amine light stabiliser (Tokuyoku Co., Ltd. melk. trade name SANPELUS-7770) weight section. Hindered phenolic antioxidant (Tokuyoku Co., Ltd. melk. trade name ISUCA NOx 1010) 1 weight section. 2) 1 weight section. 3) 5 weight section. 4) 5 weight section. 5) 5 weight section. 6) 5 weight section. 7) 5 weight section. 8) 5 weight section. 9) 5 weight section. 10) 5 weight section. 11) 5 weight section. 12) 5 weight section. 13) 5 weight section. 14) 5 weight section. 15) 5 weight section. 16) 5 weight section. 17) 5 weight section. 18) 5 weight section. 19) 5 weight section. 20) 5 weight section. 21) 5 weight section. 22) 5 weight section. 23) 5 weight section. 24) 5 weight section. 25) 5 weight section. 26) 5 weight section. 27) 5 weight section. 28) 5 weight section. 29) 5 weight section. 30) 5 weight section. 31) 5 weight section. 32) 5 weight section. 33) 5 weight section. 34) 5 weight section. 35) 5 weight section. 36) 5 weight section. 37) 5 weight section. 38) 5 weight section. 39) 5 weight section. 40) 5 weight section. 41) 5 weight section. 42) 5 weight section. 43) 5 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101427

next — as the (B) ingredient — octylic acid (2-ethylhexanoic acid) calcium salt (the Nihon Kagaku Sangyo Co. Ltd. make). The trade name NIKKAOKU Chicks Ca %o toluene solution was used as a hardening agent. Lin / for the thing for which distearyl amine (the Kao Corp. make, Firmin D80) is respectively shown in Table 1 as a (C) ingredient and which could number / of weight sections / — measure, could use the spatula, and was stirred and mixed / working example 11.

[0143]

here = a (B) ingredient of this invention = naphthoic acid vanadium (the Nihon Kagaku Sangyo Co. Ltd. make), as a trade name NIKKAMATU textile V 2% toluene solution and the (C) ingredient = disaleryl amine (the Kao Corp. make). The number of weight sections = used thing which shows Firm D88 in Table 1 respectively is made into working example 2, (B) as an ingredient = oxydic acid (2'-ethylhexanoic acid) iron (the Nihon Kagaku Sangyo Co. Ltd. make), an number of weight sections = used thing which shows respectively lauryl amine (made by Wako Pure Chemical Industries, Ltd. in Table 1 as a trade name NIKKAKU Chikdo Fe 6% toluene solution and a (C) ingredient is made into working example 3. The number of weight sections = used thing and furthermore shows respectively lauryl amine (made by Wako Pure Chemical Industries, Ltd.) in Table 1 as a (B) ingredient as oxydic acid (2'-ethylhexanoic acid) titanium (3% toluene solution). The ingredient was made into working example 4. On the other hand, the number of weight sections = used thing which shows lauryl amine in Table 1 respectively as (B) oxydic acid (2'-ethylhexanoic acid) (Japanese east transformation Make, trade name neo SUTAN U-28) and a (C) ingredient was made into the comparative example 1.

[0144]

These base resin and hardening agents were mixed by the ratio shown in Table 1, respectively, and then casted into the aluminum base material to H type in accordance with the manufacturing method of the tensile adhesive property specimen to which is specified JIS A5798-1982 was used. Care was taken that conditions were made into 23 °C ± 5 °C and 50% ± 5% RH. The used aluminum was the aluminate treatment aluminum of the 5050-2mm size based on JIS H4000 as a substrate. After purifying by methyl ethyl ketone (made by Wako Pure Chemical Industries, Ltd.) before being filled up with the compound, the primer (the Tensoluxinadine Duro Coming make, trade name) was applied to the surface of the aluminum base material with a brush and 30 min was dried at 100 °C for 30 min. The adhesive temperature for curing and 30 min was

Name: \_\_\_\_\_  
FOI # \_\_\_\_\_

[0145] In accordance with the tensile cement test method of 5758 - 1992 N of JIS A, the examination was presented with H type sample obtained by the above-mentioned method on condition of speed-of-reaction 50 mm/min, amount 23.4g and the thermostatic chamber of 50±0.5% of humidity.

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The recovery was measured by the following methods. First, after making H type sample which ended at the above-mentioned rate of health immersed into 50 °C warm water for one day and taking it out, it was placed under the room temperature on the 1st. Subsequently, after having compressed and heated to 70% so that sample thickness may be set to 12 to 8.4 mm, and heating a sample in 100 °C hot air drying equipment for one day, it released from compression and was heated under the room temperature on the 1st. The thickness of the sample in this case was measured and the recovery to compressed state was computed.

50147

Time (leather-covered time) until it fits the same compound in an ointment can simultaneously as a measure which estimates hardness and stretches a hide on the surface under 23 °\* and the conditions of 50% of relative humidity was measured. The one where leather-covered time is shorter shows that hardness is high.

[0148]

A result is shown in Table 1. The filled hardened material is that cohesive failure is shown, and CF showing the fracture state in front is a fracture state which can be searched for as a sealing material.

[0149]







試料	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
試料名	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
試料番号	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
試料名	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100
試料番号	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100

[0177] When the various carboxylic acid metal salt of working example 16-28 is used as shown in Table 4, in spite of having used the silane condensation catalyst of the non-tin series, the surface has hardened and practical hardenability was shown.

On the other hand, when the carboxylic acid zinc of the comparative example 4 was used, hardenability had not hardened five days after bad.

As mentioned above, the organic polymer which has at least one reactive silicon group in the (A) molecule, (B) Carboxylic acid calcium, carboxylic acid vanadium, carboxylic acid iron, Carboxylic acid titanium, carboxylic acid potassium, carboxylic acid barium, Carboxylic acid manganese, carboxylic acid nickel, carboxylic acid cobalt, one or more sorts of carboxylic acid metal salt chosen from a carboxylic acid zirconium, and a hardenability [ \* and others ] constituent. In spite of being a non-tin series catalyst, practical hardenability is shown, and it turns out that that hardened material physical

properties are also fitness (high elongation and high intensity).

[0179]

(Constituent using the trimethoxysilyl group as a reactive silicon group)  
If a trimethoxysilyl group uses the polyoxalkylene series polymer which exists in molecular chain terminals as a reactive silicon group, a cure rate will become large from the polymer of the synthetic example 1. The direction of a trimethoxysilyl group of this is because reactivity is larger than a methyl dimethoxy silyl group. Such a polymer is indicated to JP-H11-72480A or JP-2001-72855A. For example, the same hardenability constituent as working example 6-15 can be prepared using the polymer indicated for the example 1 of manufacture of JP-H11-72480A. The tack free time of this polymer is shorter than working example 6-15.

The same hardenability constituent as working example 6-15 can be prepared using the polymer indicated for the example 1-4 of manufacture of JP-2001-72855A. The tack free time of this constituent is shorter than working example 6-15. Therefore, a catalyst amount can be lessened, if cure time is made into the same grade and the polymer which has the above-mentioned trimethoxysilyl group will be used.

[0181]

If the mixture of the polymer which has a trimethoxysilyl group, and the polymer which has a methyl dimethoxy silyl group is used as a polymer, cure time and the physical properties of a hardened material are freely controllable. For example, the hardenability constituent of working example 6-15 can be prepared using the polymer which mixed the polymer which has a trimethoxysilyl group, and the polymer which has a methyl dimethoxy silyl group by the weight ratio of 1:10-10:1.

[0182]

Working example when the polymer which has a trimethoxysilyl group is used is shown below.

[0183]

(Synthetic example 3)  
The inside of bottom of N<sub>2</sub> atmosphere 1L autoclave. According to a composite metal complex compound catalyst. It polymerizes. The obtained polypropylene oxide. Use and to the compound number average molecular weight 17,000 and the polyoxpropylene dol 100kg of molecular-weight-distribution Mw/Mn=1.20 as 1% of gamma-isocyanate propyltrimethoxysilane (Nippon Unicar make Y-5117), and a catalyst 0.05 g of diethyl tin screw isocyanatopolyisolate imide in [ U-350 ] transomeration [ Japanese east ] is added. It reacted at 30 °C until the isocyanate group was no longer detected in it under the nitrogen air current in tank, and the reaction solution content polyoxpropylenes (A-3) by which the trimethoxysilyl group was introduced into about 80% of ends was obtained.

[0184]

(Working example 28-32)

(A) The polyoxalkylene series polymer (A-1) which has as an ingredient the methyl dimethoxy silyl group obtained in the synthetic example 1. The various carboxylic acid metal salt which is the (B) ingredients according to the formula shown in Table 5 using the polyoxalkylene series polymer (A-3) which has the trimethoxysilyl group obtained in the synthetic example 3. (C) Lauryl amine which is an ingredient was measured, respectively, the spatula was used, and it stirred and mixed for 30 seconds. It was made for all the numbers of mols of the metal atom containing the number of addition parts of the various carboxylic acid metal salt of the (B) ingredient to become almost the same here. The surface was lightly pressed down with the spatula after mixing and time (leather-covered time) until a constituent adhesion at the tip of a spatula was measured. An evaluation result is shown in Table 5.

[0185]

[Table 5]



組成 (重量部)					
(A) 成分	A-1	29	30	31	32
三アリルホスフィン酸塩 (例)	A-3	100	100	100	100
(B) 成分	三アリルホスフィン酸塩 (例)	6.5	6.4	5.8	5.5
(C) 成分	三アリルホスフィン酸塩 (例)	0.01	0.01	0.01	0.01
硬化剤 (重量部)		41.0	21.0	10.0	10.0

(1) : 2-エチルヘキサンチオールビス(トリメチルシリル) 含有率 : 5 %

(2) : 2-エチルヘキサンチオールビス(トリメチルシリル) 含有率 : 5 %

[0186]

As shown in Table 5, the polyoxalkylene series polymer (A-3, working example 30 and 32) which has a trimethoxysilyl group had the cure rate quicker than the polyoxalkylene series polymer (A-1, working example 29 and 31) which has a methoxy dimethoxy silyl group.

[0187]

The same hardenability constituent as working example 6-15 can be prepared using the polyoxalkylene series polymer (A-3) which has the trimethoxysilyl group obtained in the synthetic method of example 3. The task free time of this constituent has short cure time, and good physical properties are acquired.

[0188]

(Constituent which used the epoxy resin together)

The same hardenability constituent as working example 6-15 can be prepared using the constituent which used together reactive silicon group containing organic polymer and an epoxy resin. When this constituent is used, the manifestation of adhesive strength is quick.

[Industrial applicability]

[0189]

(A) The organic polymer which has the hydroxyl group or hydrolytic basis combined with the silicon atom, and has at least one silicon content group which can construct a bridge by forming a siloxane bond. (B) Carboxylic acid calcium, carboxylic acid vanadium, carboxylic acid iron, Carboxylic acid titanium, carboxylic acid potassium, carboxylic acid barium. The hardenability constituent containing one or more sorts of carboxylic acid metal salt which it comes to choose out of carboxylic acid manganese, carboxylic acid nickel, carboxylic acid cobalt, and a carboxylic acid zirconium. While revealing practical hardenability and stability, the hardened material obtained shows the outstanding mechanical property which has high intensity and high elongation as compared with the case where the carboxylic acid tin currently used conventionally is used as curing catalyst. Therefore, the hardenability constituent of this invention is very useful as various elastomers, such as a sealing material, adhesives, a binder.

[Translation done.]